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Influence of the composite surface structure on the peel strength of metallized carbon fibre-reinforced epoxy

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ABSTRACT

In this work, the effect of mechanical pre-treatment on the surface structure of carbon fibre-reinforced epoxy composites and on its peel strength of electroless/electroplated copper was investigated. Sandblasting with Al_2O_3 was used to pre-treat the composite surface. The parameters investigated were blasting time (3 s, 6 s and 9 s) and nozzle distance to substrate (300 mm and 500 mm). A two-step metallization process was used for depositing copper coatings on the pre-treated composite surface. First, an eletroless plating process was used to deposit a thin layer on the surface. Second, an electroplating process was used to reinforce the thickness of the coating. Increased blasting intensity leads to a significant increase in surface roughness, which promotes mechanical anchoring effects of the coating. Scanning electron microscopy images and contact angle measurements confirm the results of the surface roughness. The adhesion of sandblasted composites, characterized by measuring the peel strength, is 10 times higher compared to untreated specimens. In addition to the mechanical anchoring mechanism the exposure of carbon fibres on the surface due to the blasting process promoted a stronger bonding to copper, due to the higher, electrical conductivity of the fibres in comparison to the matrix.

1. Introduction

The usage of carbon fibre-reinforced polymers (CFRP) within the automotive and aerospace industry is continuously growing in the last decades to replace traditional materials and achieve weight reduction. Currently, this is not only the case for structural parts, but also for storage systems for cryogenic liquid hydrogen, which is gaining attention from both academic and industrial communities.

Liquid hydrogen as an energy carrier is of special interest because of the much higher gravimetric energy density compared to gaseous and solid stored hydrogen as well as compared to conventional fuel systems. Prototypes and technology demonstrators using liquid hydrogen can be found in automobile and aerospace industries [1]. Traditionally made of stainless steel, the cryogenic storage systems can alternatively be manufactured with CFRP leading to a weight reduction of approximately 60% [1].

However, standard epoxy composites are not sufficiently tight for the storage of liquid hydrogen because of the higher permeation and outgassing rates compared to stainless steel. A metal coating on the surface of the polymer composite is therefore required as permeation barrier in order to fulfill the requirements [1].

Suitable coating processes of CFRP are vacuum-metallization (e.g. PVD or CVD), indirect metallization (e.g. hot foil stamping) and plating processes (e.g. electroless/electrolytic plating) [1–3]. Hot foil stamping is a suitable and economically viable method for relatively simple 2D geometries [2]. However, it cannot be employed for the manufacture of cryogenic storage systems. In the case of these complex-shaped 3D parts manufactured with CFRP, plating process is the most suitable coating process mainly because of faster deposition rates, higher ductility of the coatings and lower process temperatures compared to PVD or CVD processes [4].

Regardless which of the above-mentioned processes is selected to coat the CFRP with a metallic layer for permeation barrier purposes, it is generally very difficult to create consistently high adhesive strength levels between the composite and the coating materials [5]. This is due to the much lower polarity of the polymer surface in comparison to the coating material [6]. As consequence of the weak adhesion, the coating can detach from the CFRP surface leading to a significant permeability increase. This process is further accelerated due to the fact that storage systems are subjected to dynamic loadings [7]. One reason for

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this arises due to the difference of the outer temperature (room temperature, e.g. 23 °C) and inner temperature (−253 °C, liquid hydrogen). Furthermore, such storage systems are subjected during their lifecycle to a number of predictable and unpredictable mechanical loadings, especially considering that these systems are employed in the transport sector. For instance, considering the promising use of cryogenic hydrogen as energy carrier for satellites, the storage system and the CFRP/coating interface must remain intact during and after the rocket launch.

To increase the adhesion of the polymer substrate with the coating layer, surfaces are often treated in a way to: a) increase the surface roughness for mechanical adhesion, or b) modify the surface energy to increase the wettability and adsorption [2,8].

In both cases the surface is modified by pre-treatment processes, which can be generally classified as mechanical, chemical or electrical pre-treatments. Mechanical processes are grinding and sandblasting whereas etching and wet-chemical surface modification are typical examples for chemical processes. Electrical pre-treatment processes include atmospheric and low-pressure plasma treatment [9].

Regarding thermoplastics, examples for chemical pre-treatment can be found in case of acrylonitrile butadiene styrene (ABS), the most widely electroless plated plastic. In this case chromic acid has two effects on the surface, which results in an improved adhesion. It increases the surface energy and wettability by oxidizing the surface and it dissolves the polybutadiene nodes in ABS, which increases the surface roughness and significantly improves the mechanical adhesion [10]. In case of polyetherimide etching with permanganate, the imide ring of the molecule is opened and allows the copper ions to be incorporated into the system, which results in a high adhesion of the cooper coating to the polymer substrate [11].

Examples for electrical pre-treatment are found in case of plasma treatment of polycarbonate surfaces for palladium chemisorption prior to electroless deposition. Charbonnier et al. showed that after plasma treatment a high efficiency in grafting chemical functions could be achieved [12]. Direct palladium chemisorption onto nitrogenated groups is highlighted. However no influence on the adhesion was presented.

Chemical pre-treatment of epoxy resins is very difficult due to the narrow processing window and the high chemical resistance of this thermoset to most etching media. This leads to difficulties achieving a structured surface, as either too long times or too aggressive media will lead to its destruction [13]. Effect of alkaline etching on the surface roughness of a fibre-reinforced epoxy composite has been studied by Roizard et al. [14]. It has been shown that a small change in the topography could be achieved but the effect of the adhesion of a metal coating was not investigated. Kirmann et al. [15] studied the effects of the alkaline permanganate etching of epoxy on the peel adhesion of electrolessly plated copper on a fibre-reinforced epoxy composite. In this study the adhesion could be controlled by chemical etching with alkaline permanganate. An extra epoxy layer was applied to the composite surface to avoid fibre damage during etching.

Electrical pre-treatment of epoxy-based composites can also be found in the literature, for instance in case of plasma surface treatment of carbon fibre-reinforced epoxy composites [16–18]. Zaldivar et al. [16] studied the effect of atmospheric plasma treatment on the chemistry, morphology and resultant bonding behavior. The bonding strength after plasma treatment could be increased as much as approximately 50%. In a further study, Zaldivar et al. [17] continued the investigation of how plasma treatment process parameters affect the surface chemistry and the bonding behavior. The changes in the surface chemistry after the plasma treatment could be correlated with the adhesive bond strength. These studies looked at epoxy bonding but did not investigate metallized surfaces.

Sandblasting and its parameters as mechanical pre-treatment have been mainly investigated on metal substrates [19,20]. It has been shown that adhesion of a coating is strongly dependent on the surface roughness of the metal substrate, which can be regulated by blasting parameters. Generally speaking, higher blasting intensity (e.g. higher blasting pressure, lower distance, higher times) leads to a higher surface roughness [21].

For epoxy composites the influence of blasting angle on the adhesion of metal coatings was studied by Menningen et al. [5]. Advantages of this pre-treatment include higher adhesion strengths in comparison to grinding and easier processing in comparison to chemical etching [2,21]. Fracture mechanics was applied to the adhesion and a change from 30° to 90° blasting angle led to an increase in energy release rate of approximately 40%. But in this study no quantitative analysis of the surface structure was presented. In a further study Menningen et al. [22] investigated the effect of micro roughening on the adhesion strength of a nickel coating on a CFRP surface. It has been shown that an increase in blasting pressure leads to higher surface roughness but not necessarily to higher adhesion strength. However the influence of further blasting parameters, such as blasting time and distance, on the surface structure and adhesion strength is surprisingly still not investigated.

There is therefore a lack of knowledge in the literature in the field of copper-plated carbon fibre-reinforced epoxy composites, which we cover with this manuscript. This study focuses on the effect of the surface structure, generated with a mechanical pre-treatment method (sandblasting), on the peel strength of copper electroless-/electroplated fibre-reinforced epoxy composites. The topography and the wettability of the surface of the composite are heavily dependent on the selected pre-treatment process and its parameters [2]. This study presents a correlation between the surface properties of the substrates and the peel strength of the metallized material as well as the parameters of the pre-treatment process.

2. Experimental

2.1. Substrate material

In this study CFRP material consisting of carbon fibres (non-woven 0°/90° biaxial NCF HS Carbon from WELA) with an areal weight of 300 g/m^2 and a toughened epoxy resin as matrix (XU3508/XB3486 from Huntsman) were used. The CFRP laminates were manufactured by VARTM-process in a 1-part machine setup with a two-sided hard mould. The application of release agent Loctite Frekote 770-NC was done thoroughly on the mould surfaces as mould preparation before injection. The laminate thickness of 2 mm corresponds to a fibre volume content of approximately 50%. The laminates were cured at 100 °C for 5 h according to the resin manufacturer's datasheet.

2.2. Surface pre-treatment

The CFRP surface must be pre-treated prior to metallization of the material. The method investigated in this study is sandblasting with aluminum oxide and 200 – 300 μm grit size and a mohs hardness of 10. The parameters investigated are blasting time (3 s, 6 s and 9 s) and nozzle distance to substrate (300 mm and 500 mm). The depth of abrasion is dependent on the blasting time whereas the nozzle distance influences the blasting medium velocity and thus the kinetic energy of a blasting particle. The sandblasting machine ST 1200 ID-Z-SB with a die diameter of 10 mm is used to perform the tests. Constant parameters are blasting pressure of 2 bars and a blasting angle of 90°. All plates including the reference laminate were cleaned using an ultrasonic bath with equal parts of ethanol and water for 30 min at 25 °C prior to the coating process.

2.3. Mechanical properties of the untreated and pre-treated composite

The mechanical properties of the untreated and sandblasted composites were investigated under quasi-static 3-point bending using a universal testing machine Zwick Z2.5. For the sandblasted composites flexural properties of specimens exposed to the highest blasting

intensity were investigated. The test was carried out according to EN ISO 14125 using a rectangular bar horizontally positioned on two supports. The specimens were subjected to a vertical force applied midway between the supports at a velocity of 2 mm/min. The specimens were prepared according to class IV in the standard with the dimensions 100 mm × 15 mm. For each of the two series five measurements were performed in order to emphasize the repeatability of the results.

2.4. Coating process

The CFRP substrates were coated by the electroless/electrolytical plating process. Direct electrolytical plating of CFRP is impossible due to the electrical insulation of the polymer matrix. On account of this, a thin adherent conductive layer was chemically deposited on the CFRP surface. For this chemical deposition, the surface is made electrically conductive with a one-step activator to leave as many palladium ions on the surface as possible. Therefore the substrate was dipped into an aqueous solution consisting of a stabilized Pd–Sn colloid. Palladium needs to be protected in order to prevent agglomeration and drop out [23]. In an accelerator bath the enclosed Pd ions are broken free to leave palladium on the surface. At molecular level, the single palladium atoms are not homogeneously dispersed on the surface but they create clusters of molecular size. However they are packed enough in order to provide a homogeneous Cu layer. After this activating process, a 1 μm thick copper coating was deposited electrolessly on the surface and finally electrolytically plated with the same coating material. The final coating thickness was at least 40 μm. A rigorous surface preparation procedure was employed in this study. The electroless-/electroplating process is shown below including further cleaning steps which are as well applied to all plates:

1 Acidic Cleaner Circuposit™ 3323A for 5 min at 50 °C
2 Rinsing in deionized water for 1 min at RT
3 Low concentrated etching sulphuric etching for 1.5 min at 35 °C
4 Rinsing in deionized water for 1 min at RT
5 Pre-Dip Circuposit™ 3340 for 0.5 min at RT
6 Activator (solution of colloidal palladium–tin) for 4 min at 40 °C
7 Rinsing in deionized water for 1 min at RT
8 Accelerator (aqueous solution of fluoroboric acid) for 6 min at RT
9 Rinsing in deionized water for 1 min at RT
10 Electroless plating of Cu (aqueous solution of copper sulphate, ethylenediaminetetraacetic acid, sodium hydroxide, formaldehyde and sodium cyanide) for 20 min at 45 °C
11 Rinsing in deionized water for 1 min at RT
12 Pickling for 0.5 min at RT
13 Electroplating of Cu (aqueous solution of copper sulphate, sulphuric acid and sodium chloride) for 20 min at 1.8 A/dm^2
14 Rinsing in deionized water for 1 min at RT
15 Drying for 13 min at 65 °C

Table 1
Surface roughness and contact angle of a blasted carbon fibre-reinforced epoxy composite as a function of different blasting parameters.

Parameters		R_a (μm)	R_z (μm)	Contact angle (deg)
Distance (mm)	Time (s)			
Reference		0.30 ±0.07	2.94 ±1.05	106 ±5
300	3	1.56 ±0.26	17.68 ±3.41	141 ±12
	6	2.96 ±0.27	26.32 ±1.88	139 ±11
	9	5.33 ±0.61	37.81 ±3.76	135 ±7
500	3	1.16 ±0.29	12.74 ±3.50	111 ±5
	6	2.69 ±0.71	23.86 ±4.12	139 ±11
	9	3.84 ±0.42	33.28 ±4.15	140 ±4

2.5. Surface structure

2.4.1. Surface roughness

The roughness measurements were carried out with a Universal Surface Tester 100 from Innowep GmbH.

A 60° steel cone with a radius of curvature of 30 μm was used as tip to measure the surface profile and roughness of the pre-treated samples. A constant tip force of 1 mN and a tip speed of 0.1 mm/min were set to ensure reproducible roughness measurements according to DIN EN ISO 4287 and ASTM D 7127 – 05. The surface of the samples were measured by 10 lines with a parallel distance of 2 mm and a measuring length of 20 mm in order to obtain representative information about the roughness.

2.4.2. Microscopy

Surface investigations of the CFRP substrates were carried out by light and electron microscopy.

An optical microscope, Keyence VHX 100, was used, to look at the deposits on the interfacial side of the coatings and on the substrates after the mechanical testing of the adhesion strength.

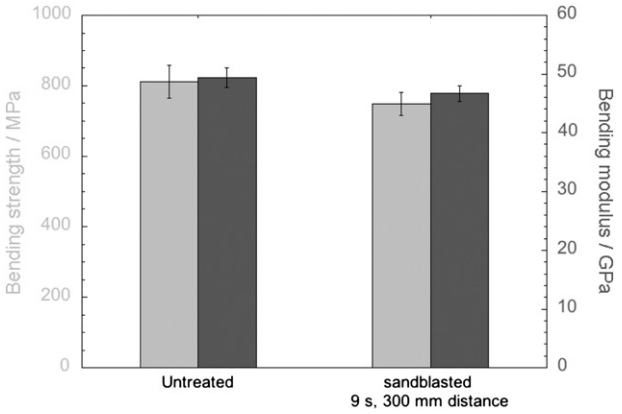

Fig. 1. 3-point bending test of an untreated and sandblasted CFRP.

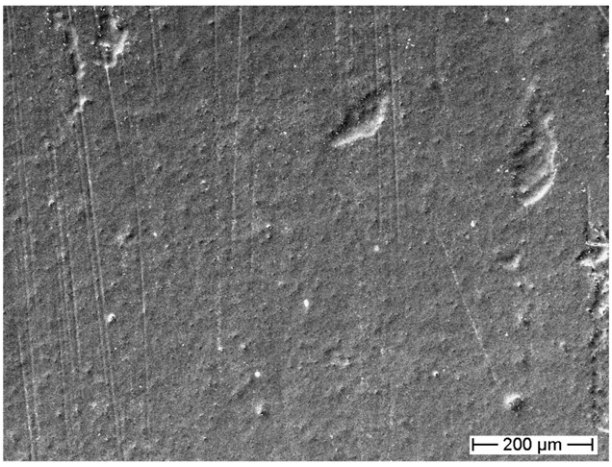

Fig. 2. SEM image of an untreated CFRP substrate.

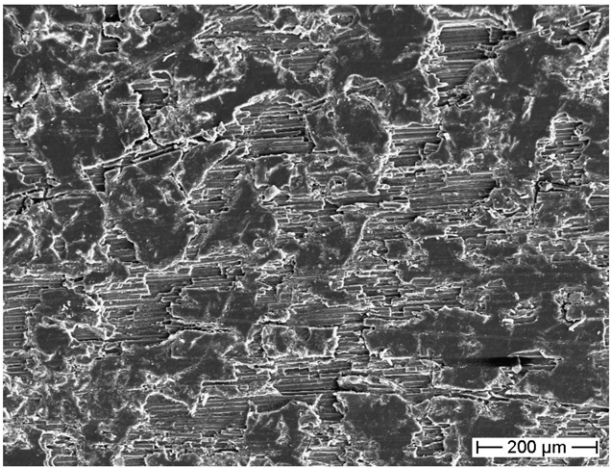

Fig. 3. SEM image of an Al_2O_3 blasted CFRP substrate (time 3 s, distance 500 mm).

A scanning electron microscope (SEM), Jeol JSM-IC 848, was used to inspect the topography of the untreated and pre-treated CFRP surfaces. The samples were gold sputtered prior to the SEM investigation.

2.4.3. Contact angle and surface energy

In measuring the contact angle the surface tension of pre-treated samples and consequently the degree of wettability are determined. A drop of fluid with a defined volume and known surface tension is applied on the sample surface. The contact angle θ is then measured in the three-phase system solid (S), liquid (L) and gaseous (G) [2].

Contact angles of test liquids were recorded using a goniometer. The liquids tested were distilled water, and diiodomethane. Five droplets of each liquid, 2 µl in volume, were measured on each samples surface, in order to measure the contact angle. For each sample, the mean and standard deviation were calculated. Using the Owens, Wendt, Rabel, and Kaeble method, surface energy values were calculated as follows [24]:

$$\frac{(1+\cos\theta)\cdot\gamma_L}{\sqrt{\gamma_L^d}} = \sqrt{\gamma_s^p}\cdot\sqrt{\frac{\gamma_L^p}{\gamma_L^d}} + \sqrt{\gamma_s^d}$$

θ is the static contact angle of the liquid on the surface of the polymer, γ_L is the surface energy of liquid and is taken from the literature [25] and γ_s is the surface energy of the polymer. Superscripts d and p represent the dispersive and polar components of the surface energy accordingly. It should be noted that the total surface energy of a liquid or solid is equal to the sum of the dispersive and polar components.

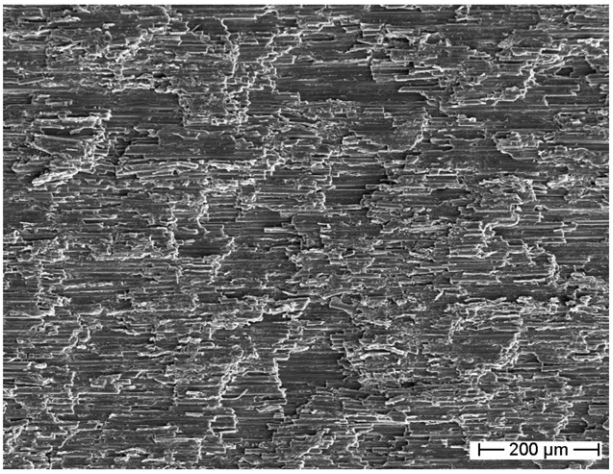

Fig. 4. SEM image of an Al_2O_3 blasted CFRP substrate (time 6 s, distance 500 mm).

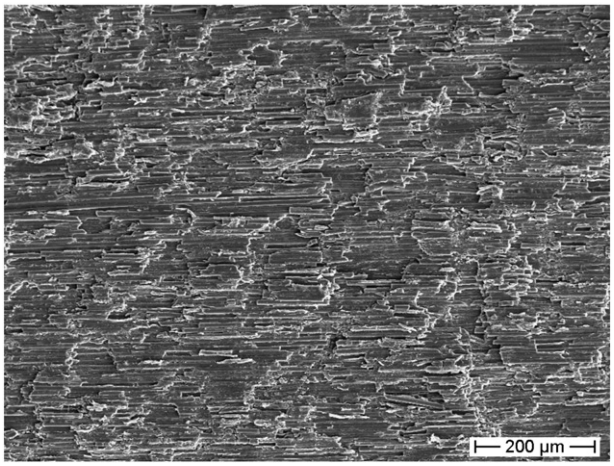

Fig. 5. SEM image of an Al_2O_3 blasted CFRP substrate (time 9 s, distance 500 mm).

2.5. Peel strength

The peel test was carried out according to ASTM B 533-85 using a universal testing machine, Zwick Z2.5. A 25 mm wide metal stripe was cut out of the substrate, using a paper knife, torn off at one end and peeled off at a velocity of 25 mm/min. The force was recorded as a function of the measuring path by the software. To calculate the peel strength the mean of the recorded force was used and divided by the width of the peeled stripes. The peeling orientation was parallel to the fibres. The testing standard used does not provide any specific orientation for the specimens. Some unpublished investigations of our group comparing pulling orientation 0° and 45° did not show any difference in peel strength.

3. Results and discussion

In order to assure that all composite plates had a sufficient curing and the same conditions for further processing steps, DSC measurements according to ISO 14322 were performed after production of the laminates, surface pre-treatment and coating process. After the initial curing cycle the system had a degree of cure above 98% and no further change was observed after the subsequent steps.

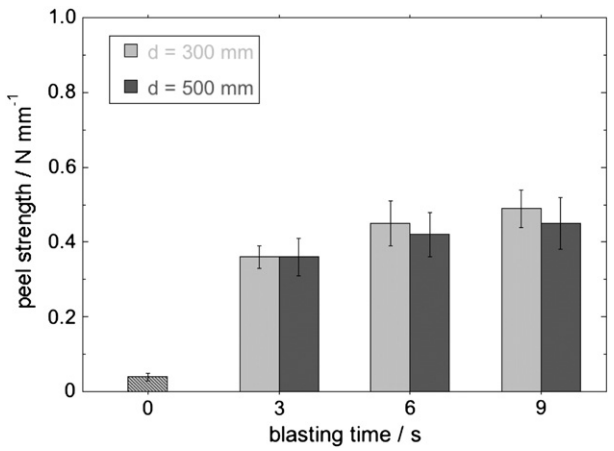

Fig. 6. Peel strength as a function of blasting time and nozzle distance d.

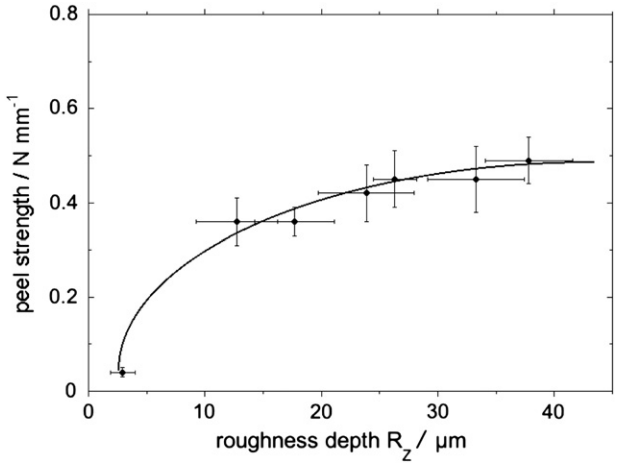

Fig. 7. Peel strength as a function of roughness depth R_Z.

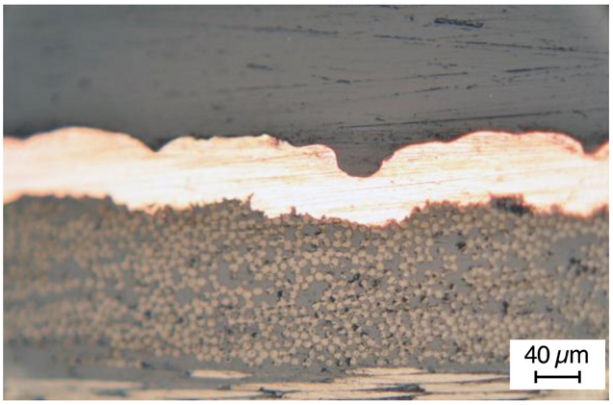

Fig. 9. Cross section of copper-coated CFRP substrate after sandblasting with Al_2O_3 (500 mm and 6 s).

3.1. Surface structure after pre-treatment

The pre-treatment process influences the surface structure of the composite and consequently the topography of the substrates. Especially the surface roughness is an important aspect in correlating the adhesion strength to the topography of the substrates.

Due to the manufacturing process, the CFRP laminates exhibited a closed epoxy matrix layer at the surfaces. Therefore, it is possible to pre-treat the samples within the epoxy matrix layer on the one hand and on the other hand to remove the outer layer while carbon fibres become exposed to some extent. Damage of the fibres can be expected and can lead to lower mechanical properties. In order to investigate this aspect, the mechanical properties of the untreated and sandblasted composites were also investigated under quasi-static 3-point bending. Fig. 1 shows the results of the bending properties of an untreated specimen and after sandblasting for 9 s and 300 mm distance. A minor performance loss of approximately 5% was observed after sandblasting. The fatigue properties of the mentioned materials (including low temperature cycling) are currently under investigation and will be the topic of a subsequent manuscript.

The variation of the blasting parameters time and distance results in a significant increase in surface roughness. Table 1 shows the mean roughness index, the roughness depth and the contact angle of sand blasted carbon fibre-reinforced epoxy composites as a function of the blasting parameters. As expected, it is clearly visible that the surface roughness is increased at higher blasting times and at a reduced distance. The mean roughness index R_a is approx. 18 times higher in case of the longest blasting time and smallest distance than the untreated reference sample. On the other hand the increase at a distance of 500 mm compared to 300 mm did not show a major effect on the roughness as the particles experience only a minor kinetic energy loss when traveling such short distances (200 mm) in air.

The effect of mechanical pre-treatment on the surface structure of carbon fibre-reinforced epoxy composite substrates is also seen when comparing the contact angles of untreated and blasted samples. The untreated sample shows low wettability referring to the contact angle of approximately 102°. After blasting with Al_2O_3 the contact angle increases and does not further significantly change at longer blasting times in case of a blasting distance of 300 mm. A significant change is merely noticeable between 3 s and 6 s at a distance of 500 mm. The increase in the contact angle can be attributed to the capillary depression effect. With increased surface roughness the adhesion properties decrease because the fluid is not pulled into the cavities but remains on top of the embossment of the surface.

Sandblasting of the CFRP samples generates a non-uniform surface structure characterized by dimples and furrows. The difference of an untreated and pre-treated sample surface is clearly visible in Figs. 2 to 5. At longer blasting times more epoxy resin of the outer layer is removed so that the carbon fibres are also exposed and damaged (Fig. 5). According to Table 1, the depth of the abrasion and the wastage rate seems to be lower at a distance of 500 mm compared to a distance of 300 mm. The influence of the blasting time and distance shown in the SEM images is reflected in the surface roughness measurements. Comparing the untreated specimen to the material blasted for 3 s a major change on surface roughness can be observed. However, longer times, namely 6 and 9 s only led to minor changes in surface roughness.

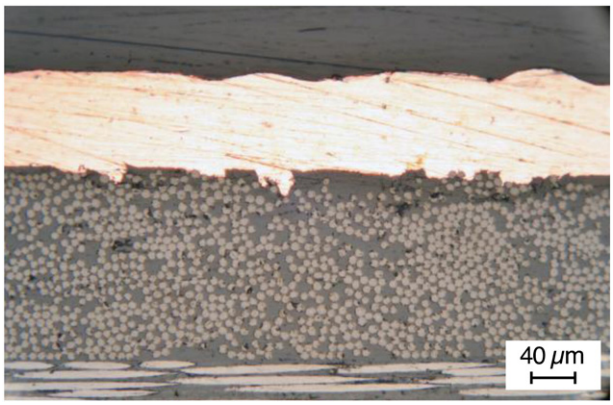

Fig. 8. Cross section of copper-coated CFRP substrate after sandblasting with Al_2O_3 (500 mm and 3 s).

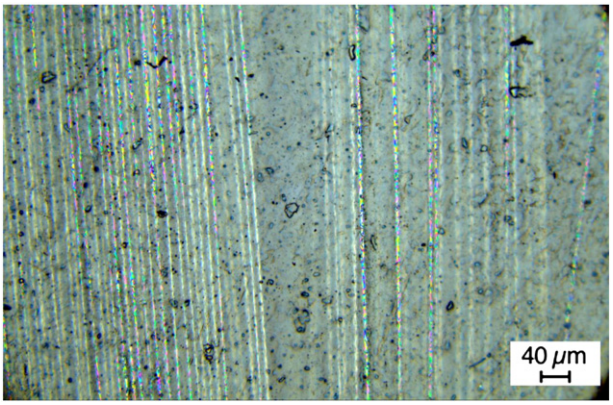

Fig. 10. Fracture surface of an untreated CFRP substrate after peel test under optical microscopy.

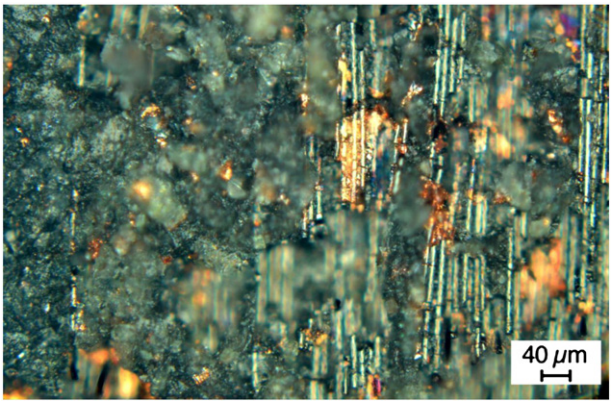

Fig. 11. Fracture surface of an Al_2O_3 blasted CFRP substrate after peel test under optical microscopy (blasting time 9 s, blasting distance 500 mm). Amplification magnitude: 20×.

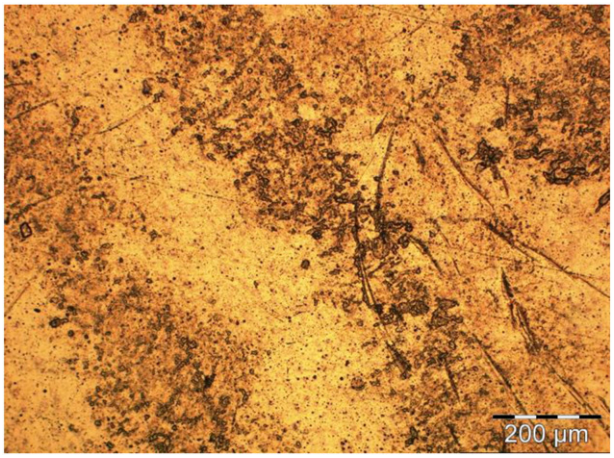

Fig. 13. Fracture surface of the back side of a Cu layer of an untreated CFRP substrate after peel test under optical microscopy.

3.2. CFRP/Metal layer adhesion

The force required to separate a metallic coating from its polymer substrate is determined by the interaction of several factors: the components and quality of the polymer moulding compound, the moulding process, the process used to prepare the substrate for electroplating, and the thickness and mechanical properties of the metallic coating. If only one of the parameters is changed at time, peel strength can be used to quantify the influence of this parameter on the adhesion strength. Fig. 6 shows the influence of the blasting time and distance on the peel strength of copper-coated CFRP substrates. As expected, an increase of the roughness leads to interlocking effects at the surface and consequently higher peel strength. It can clearly be seen that blasting time plays a role on the peel strength, whilst no significant difference is observed between the two blasting distances investigated here. The peel strength of blasted samples, even for 3 s blasting time, is approximately 10 times higher than that of untreated samples. An increase of the blasting time, which leads to a rougher surface, does not lead to a major further increase of the peel strength though. This behavior is seen in Fig. 7. One explanation for that is the fact that the copper coating has a minimum thickness of only 40 µm, and by reaching a substrate roughness around this value interfacial weak points are formed. This can be seen in Figs. 8 and 9 where cross-section images of sandblasted and copper-coated CFRP substrates are shown. The increase in surface roughness changes the appearance of the coating, as the metallization process is kept constant. Comparing these two figures one can see that in the case of the sandblasted metallized substrate with a higher blasting intensity the copper layer is in direct contact with the carbon fibres, while in the sandblasted composite with a lower blasting intensity the copper is mostly at the top of the resin-rich zones. As there was no significant difference in the images between blasting time 6 and 9 s, only one image is presented here. The exposure of carbon fibres due to sandblasting was previously observed and described (Figs. 3 to 5). This aspect should also be taken into account when considering the increase of the peel strength observed in this study. Due to their higher electrical conductivity, carbon fibres exposed at the surface promote the deposition of electroplated copper on the surface on a more readily and stronger way than the non-conductive epoxy layer. This leads to a local stronger bonding force between the layer and the copper, resulting in higher peel strength. This behavior can be observed looking at the fracture surfaces of the peel-strength-specimens, Figs. 10, 11 and 12.

Fig. 10 shows the surface of an untreated CFRP surface, which is featureless, indicating a very low adhesion between substrate and copper. On the other hand, copper residues can be observed at the fracture surfaces of the blasted specimens. Moreover, these residues are located directly over the carbon fibres, which were exposed after the blasting treatment. This demonstrates the stronger affinity between the high electrical conductive carbon fibres and copper in comparison to the epoxy/copper interface. The surface of the peeled metal coating was observed using light microscopy. The back side of copper layers removed from the substrate surface after peel test are seen in Figs. 13 and 14. Fig. 13 shows the back side of a Cu layer of an untreated CFRP surface whereas in Fig. 14 a Cu layer back side of a sandblasted CFRP surface (9 s, 500 mm) is visible. There is no presence of fibres or resin debris on the peeled metal. The specimens

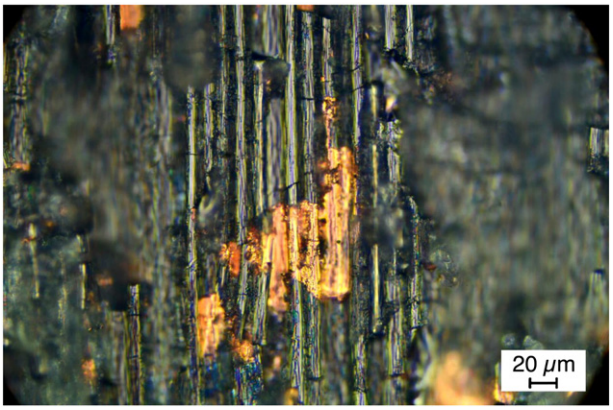

Fig. 12. Fracture surface of an Al_2O_3 blasted CFRP substrate after peel test under optical microscopy (blasting time 9 s, blasting distance 500 mm). Amplification magnitude: 50×.

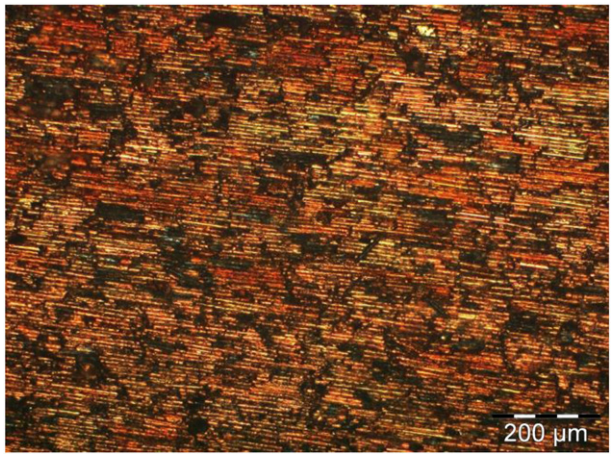

Fig. 14. Fracture surface of the back side of a Cu layer of an Al_2O_3 blasted CFRP substrate after peel test under optical microscopy (blasting time 9 s, blasting distance 500 mm).

with higher adhesion present simply a metal coating, which copies the surface structure of the substrate (Fig. 14). In such cases there is evidence of cohesive failure of the coating (as cooper debris are present on the substrate) but there is no evidence of cohesive failure of the laminate.

4. Conclusions

In this work, the effect of mechanical pre-treatment on the surface structure of carbon fibre-reinforced epoxy composites and on its peel strength of electroless/electrodeposited copper was investigated. The pre-treatment method employed here was sandblasting with Al_2O_3, where blasting time and distance were changed. An impressive 10-fold increase of the peel strength was obtained in this study.

On one hand, this increase was due to the higher surface roughness of the substrate after pre-treatment. Simultaneously, the contact angle also increased due to the capillary depression effect. The roughness was mainly influenced by the blasting time and to a lower extent by the blasting distance.

On the other hand, the peel strength of the blasted specimens also increased due to the exposure of carbon fibres at the surface. Due to the higher electrical conductivity of carbon fibres in comparison to epoxy, the electroplating process was locally facilitated and a stronger adhesion of the copper coating to the exposed carbon fibres could be reached. To our best knowledge this is the first time that such behavior is reported in the literature, and leads to new possibilities and approaches for the development of future modern metallized CFRP.

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